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J. Serb. Chem. Soc. 88 (2) S35–S50 (2023) *Supplementary material*

SUPPLEMENTARY MATERIAL TO

Divergent synthesis and antitumour activity of novel conformationally constrained (–)-muricatacin analogues

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PHYSICAL AND SPECTRAL DATA OF SYNTHESIZED COMPOUNDS

*Methyl-(Z)-3-O-methyl-5,6-dideoxy-1,2-O-isopropylidene-α-d-xylo-hept-5-enofuranuronate (**4**)*

Colourless oil $[\alpha]_D$=−143 (*c* 0.5, MeOH), R_f=0.31 (3:2 PE/Et_2O). IR ($CHCl_3$): ν_{max} 1721 (C=O), 1165 (O-C, ester). ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.20 and 1.39 (2 × s, 3 H each, C*Me*$_2$), 3.22 (s, 3 H, OCH_3), 3.61 (s, 3 H, CO_2CH_3), 3.92 (d, 1 H, $J_{3,4}$=3.3 Hz, H-3), 4.48 (d, 1 H, $J_{1,2}$=3.9 Hz, H-2), 5.51 (ddd, 1 H, $J_{4,5}$=6.9, $J_{3,4}$=3.3, $J_{4,6}$=1.6 Hz, H-4), 5.82 (dd, 1 H, $J_{5,6}$=11.8, $J_{4,6}$=1.7 Hz, H-6), 5.82 (d, 1 H, $J_{1,2}$=3.9 Hz, H-1), 6.19 (dd, 1 H, $J_{5,6}$=11.8, $J_{4,5}$=6.9 Hz, H-5). ^{13}C NMR (100 MHz, $CDCl_3$): δ 26.2 and 26.8 (2×CH_3-isopropylidene), 51.3 (CO_2CH_3), 58.0 (OCH_3), 77.6 (C-4), 82.2 (C-2), 86.2 (C-3), 104.9 (C-1), 111.5 (Me_2C), 120.6 (C-6), 145.2 (C-5), 165.8 (CO_2CH_3). HRMS-Heated ESI-Orbitrap: *m/z* 281.09908 (M^++Na), calcd. for $C_{12}H_{18}NaO_6$: 281.10011; *m/z* 297.07278 (M^++K), calcd. for $C_{12}H_{18}KO_6$: 297.07404.

*Methyl-(E)-3-O-methyl-5,6-dideoxy-1,2-O-isopropylidene-α-d-xylo-hept-5-enofuranuronate (**5**)*

White crystals, mp 47 ºC (Et_2O/hexane), $[\alpha]_D$=−59.6 (*c* 0.5, $CHCl_3$), R_f =0.20 (3:2 PE/Et_2O). IR ($CHCl_3$): ν_{max} 1724 (C=O), 1166.8 (O-C, ester). ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.34 and 1.51 (2 × s, 3 H each, C*Me*$_2$), 3.39 (s, 3 H, OCH_3), 3.75 (s, 3 H, CO_2CH_3), 3.78 (d, 1 H, $J_{3,4}$=3.2 Hz, H-3), 4.62 (d, 1 H, $J_{1,2}$=3.8 Hz, H-2), 4.80 (ddd, 1 H, $J_{4,5}$=4.9, $J_{3,4}$=3.0, $J_{4,6}$=1.7 Hz, H-4), 5.96 (d,1 H, $J_{1,2}$=3.8

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Hz, H-1), 6.18 (dd, 1 H, $J_{5,6}$=15.7, $J_{4,6}$=1.7 Hz, H-6), 6.97 (dd, 1 H, $J_{5,6}$=15.7, $J_{4,5}$=4.9 Hz, H-5). ^{13}C NMR (100 MHz, $CDCl_3$): δ 26.1 and 26.8 (CMe_2), 51.6 (CO_2CH_3), 58.2 (OCH_3), 79.3 (C-4), 81.9 (C-2), 85.6 (C-3), 104.8 (C-1), 111.8 (Me_2C), 122.7 (C-6), 141.4 (C-5), 166.4 (-CO_2CH_3). HRMS-Heated ESI-Orbitrap: *m/z* 281.09931 (M^++Na), calcd. for $C_{12}H_{18}NaO_6$: 281.10011; *m/z* 297.0735 (M^++K), calcld. for $C_{12}H_{18}KO_6$: 297.07404.

*Dimethylacetal 2,5-anhydro-6-deoxy-3-O-methyl-l-ido-hepturono-4,7-lactone (**6**)*

Colorless oil; $[\alpha]_D$=−11 (*c* 0.5, $CHCl_3$), R_f=0.26 (4:1 $CHCl_3$/EtOAc). IR ($CHCl_3$): ν_{max} 1789 (C=O). ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.70 (d, 1 H, $J_{6a,6b}$=18.8 Hz, H-6a), 2.74 (dd, 1 H, $J_{6a,6b}$=18.8, $J_{5,6b}$=4.2 Hz, H-6b), 3.42 (s, 3 H, OCH_3, C-3), 3.44 i 3.47 (2 × s, 3 H each, OCH_3), 3.99 (d, 1 H, $J_{2,3}$=3.6 Hz, H-3), 4.06 (dd, 1 H, $J_{1,2}$=7.3, $J_{2,3}$=3.7 Hz, H-2), 4.57 (d, 1 H, $J_{1,2}$=7.3 Hz, H-1), 4.90 (d, 1 H, $J_{4,5}$=4.5 Hz, H-4), 4.97 (m, 1 H, $J_{4,5}$=4.5, $J_{5,6b}$=4.3 Hz, H-5).^{13}C NMR (100 MHz, $CDCl_3$): δ 36.0 (C-6), 53.6 and 55.2 (2 × OCH_3), 58.6 (OCH_3 C-3), 77.5 (C-5), 79.6 (C-2), 83.4 (C-3), 84.5 (C-4), 102.0 (C-1), 175.2 (C=O). HRMS-Heated ESI-Orbitrap: *m/z* 255.08469 (M^++Na), calcd. for $C_{10}H_{16}NaO_6$: 255.08446.

*3,6-Anhydro-2-deoxy-5-O-methyl-l-ido-heptono-1,4-lactone (**8**)*

White, needle-like crystals, mp 78−80 °C (EtOAc/hexane), $[\alpha]_D$=−10.2, (*c* 0.5, $CHCl_3$), R_f= 0.2 (3:2 CH_2Cl_2/EtOAc). IR ($CHCl_3$) ν_{max} 3455 (OH), 1781 (C=O). ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.14 (bs, 1 H, OH), 2.68 (dd, 1 H, $J_{2a,2b}$=18.8, $J_{2a,3}$=1.2 Hz, H-2a), 2.76 (dd, 1 H, $J_{2a,2b}$=18.8, $J_{2b,3}$=5.7 Hz, H-2b), 3.51 (s, 3 H, OCH_3), 3.82 (dd, 1 H, $J_{7a,7b}$=12.1, $J_{6,7a}$=4.4 Hz, H-7a), 3.88 (dd, 1 H, $J_{7a,7b}$ =12.1, $J_{6,7b}$=4.8 Hz, H-7b), 4.11 (d, 1 H, $J_{5,6}$=4.9 Hz, H-5), 4.23 (q, 1 H, $J_{5,6}$=4.7, $J_{6,7}$=4.7, $J_{6,7b}$=4.7 Hz, H-6), 4.95 (d, 1 H, $J_{3,4}$=4.7 Hz, H-4), 5.01 (m, 1 H, $J_{2a,3}$=1.2, $J_{2b,3}$=5.8, $J_{3,4}$=4.7 Hz, H-3). ^{13}C NMR (100 MHz, $CDCl_3$): δ 36.0 (C-2), 58.6 (-OMe), 61.5 (C-7), 76.9 (C-3), 80.5 (C-6), 85.1 (C-5), 85.3 (C-4), 175.2 (C=O). HRMS-Heated ESI-Orbitrap: *m/z* 211.05787 (M^++Na), calcd. for $C_8H_{12}NaO_5$: 211.05824.

*3,6-Anhydro-2-deoxy-5-O-metyl-7-O-nonyl-l-ido-heptono-1,4-lactone (**9**)*

Colorless, glassy crystals, mp 38–41 ºC (CH_2Cl_2/hexane), $[\alpha]_D$ = –12.8 (*c* 0.45, $CHCl_3$); R_f=0.23 (1:1 PE/Et_2O). IR ($CHCl_3$): ν_{max} 1787.89 (C=O). ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.89 (t, 3 H, *J* = 6.9 Hz, CH_3 from side chain), 1.24–1.33 (m, 12 H, 6 × CH_2 from side chain), 1.59 (m, 2 H, $OCH_2CH_2(CH_2)_6CH_3$), 2.70 (dd, 1 H, $J_{2a,2b}$=19.0, $J_{2a,3}$=2.3 Hz, H-2a), 2.76 (dd, 1 H, $J_{2a,2b}$=18.8, $J_{2b,3}$=4.9 Hz, H-2b), 3.40–3.54 (m, 5 H, OCH_3 and $OCH_2(CH_2)_7CH_3$), 3.60 (dd, 1 H, $J_{7a,7b}$=10.3, $J_{6,7a}$= 6.3 Hz, H-7a), 3.65 (dd, 1 H, $J_{7a,7b}$=10.3, $J_{6,7b}$=4.7 Hz, H-7b), 4.00 (bd, 1 H, $J_{5,6}$=4.1 Hz, H-5), 4.25 (dt, 1 H, $J_{5,6}$=4.4, $J_{6,7b}$=4.5, $J_{6,7a}$=6.3 Hz, H-6), 4.94 (dd, 1 H, $J_{3,4}$=4.8, $J_{4,5}$=0.8 Hz, H-4), 4.97 (m, 1 H, $J_{2a,3}$=2.3, $J_{2b,3}$=4.8,

$J_{3,4}$=4.8 Hz, H-3). ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.1 (CH_3), 22.7, 26.1, 29.3, 29.5, 29.6, 29.6, 31.9 (7×CH_2 from side chain), 36.0 (C-2), 58.6 (OCH_3), 68.4 (C-7), 71.8 ($OCH_2(CH_2)_7CH_3$), 76.7 (C-3), 79.6 (C-6), 83.9 (C-5), 84.9 (C-4), 175.4 (C=O). HRMS-Heated ESI-Orbitrap: *m/z* 337.19785 (M^+ + Na), calcd. for $C_{17}H_{30}NaO_5$: 337.19854.

*3,6-Anhydro-2-deoxy-5-O-metyl-7-O-octyl-l-ido-heptono-1,4-lactone (**10**)*

White cristals, mp 37–39 °C (Et_2O/hexane), $[\alpha]_D$ = –17.2 (*c* 0.5, $CHCl_3$); R_f=0.15 (7:3 PE/Et_2O). IR ($CHCl_3$): ν_{max} 1791.06 (C=O). ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.89 (t, 3 H, *J*=6.9 Hz, CH_3), 1.22–1.36 (m, 10 H, 5 × CH_2 from side chain), 1.52–1.64 (m, 2 H, $OCH_2CH_2(CH_2)_5CH_3$), 2.70 (dd, 1 H, $J_{2a,2b}$=18.9, $J_{2a,3}$=2.1 Hz, H-2a), 2.78 (dd, 1 H, $J_{2a,2b}$=18.9, $J_{2b,3}$ = 4.9 Hz, H-2b), 3.39–3.53 (m, 2 H, $OCH_2(CH_2)_6CH_3$), 3.47 (s, 3 H, CH_3 from OCH_3), 3.59 (dd, 1 H, $J_{7a,7b}$=10.3, $J_{6,7a}$ =6.4 Hz, H-7a), 3.64 (dd, 1 H, $J_{7a,7b}$=10.3, $J_{6,7b}$=4.8 Hz, H-7b), 3.99 (bd, 1 H, $J_{5,6}$=4.0 Hz, H-5), 4.25 (dt, 1 H, $J_{6,7a}$=6.3, $J_{6,7b}$=4.6, $J_{5,6}$=4.4 Hz, H-6), 4.94 (dd, 1 H, $J_{3,4}$=4.8, $J_{4,5}$=0.8 Hz, H-4), 4.97 (td, 1 H, $J_{2a,3}$=2.2, $J_{2b,3}$=4.9, $J_{3,4}$=4.9 Hz, H-3).^{13}C NMR (100 MHz, $CDCl_3$): 14.1 (CH_3), 22.6, 26.1, 29.2, 29.4, 29.6, 31.8 (6 × CH_2 from side chain), 36.0 (C-2), 58.6 (OMe), 68.4 (C-7), 71.8 [$OCH_2(CH_2)_6CH_3$], 76.8 (C-3), 79.6 (C-6), 83.9 (C-5), 84.9 (C-4); 175.4 (C=O). HRMS-Heated ESI-Orbitrap: *m/z* 323.18393 (M^+ + Na), calcd. for $C_{16}H_{28}NaO_5$: 323.18289.

*3,6-Anhydro-2-deoxy-7-O-heptyl-5-O-metyl-l-ido-heptono-1,4-lactone (**11**)*

White, needle-like cristals, mp 44−45 °C (Et_2O/hexane), $[\alpha]_D$=−10.0 (*c* 0.5, $CHCl_3$), R_f=0.16 (3:2 PE/Et_2O). IR ($CHCl_3$) ν_{max} 1791 (C=O).^{1}H NMR (400 MHz, $CDCl_3$): δ 0.86 (t, 3 H, *J*=6.9 Hz, CH_3 from side chain), 1.22−1.33 (m, 8 H, 4×CH_2 from side chain), 1.56 (m, 2 H, $OCH_2CH_2(CH_2)_4CH_3$), 2.66 (dd, 1 H, $J_{2a,2b}$=18.8, $J_{2a,3}$=1.6 Hz, H-2a), 2.73 (dd, 1 H, $J_{2a,2b}$=18.8, $J_{2b,3}$=5.3 Hz, H-2b), 3.37−3.55 (m, 2 H, $OCH_2(CH_2)_5CH_3$), 3.46 (s, 3 H, OCH_3), 3.56 (dd, 1 H, $J_{7a,7b}$=10.3, $J_{6,7a}$=6.4 Hz, H-7a), 3.61 (dd, 1 H, $J_{7a,7b}$=10.3, $J_{6,7b}$=4.8 Hz, H-7b), 3.96 (d, 1 H, $J_{5,6}$=4.0 Hz, H-5), 4.22 (dt, 1 H, $J_{5,6}$=4.4, $J_{7b,6}$=4.5, $J_{6,7a}$=6.3 Hz, H-6), 4.91 (d, 1 H, $J_{3,4}$=5.0 Hz, H-4), 4.94 (m, 1 H, $J_{2a,3}$=1.8, $J_{2b,3}$=5.0, $J_{3,4}$=5.0 Hz, H-3).^{13}C NMR (100 MHz, $CDCl_3$): δ 14.1 (CH_3), 22.6, 26.0, 29.1, 29.6, 31.8 (5 × CH_2 from side chain), 36.0 (C-2), 58.6 (OCH_3), 68.4 (C-7), 71.8 [$OCH_2(CH_2)_5CH_3$], 76.8 (C-3), 79.5 (C-6), 83.9 (C-5), 84.9 (C-4), 175.4 (C=O). HRMS-Heated ESI-Orbitrap: *m/z* 309.16716 (M^++Na), calcd. for $C_{15}H_{26}NaO_5$: 309.16779.

*3,6-Anhydro-2-deoxy-7-O-hexyl-5-O-metyl-l-ido-hexyl-1,4-lactone (**12**)*

White crystals, mp 55 °C, (Et_2O/hexane); $[\alpha]_D$=−13.2 (*c* 0.5, $CHCl_3$), R_f=0.26 (7:3 PE/Et_2O). IR ($CHCl_3$) ν_{max} 1790 (C=O). ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.90 (t, 3 H, *J*=6.9 Hz, CH_3 from side chain), 1.23–1.39 (m, 6 H, 3 ×

CH_2 from side chain), 1.60 (m, 2 H, $OCH_2CH_2(CH_2)_3CH_3$), 2.71 (dd, 1 H, $J_{2a,2b}$=18.8, $J_{2a,3}$=2.2 Hz, H-2a), 2.76 (dd, 1 H, $J_{2a,2b}$=18.8, $J_{2b,3}$=4.9 Hz, H-2b), 3.41-3.54 (m, 5 H, OCH_3 i $OCH_2(CH_2)_4CH_3$), 3.60 (dd, 1 H, $J_{7a,7b}$=10.3, $J_{6,7a}$=6.3 Hz, H-7a), 3.65 (dd, 1 H, $J_{7a,7b}$=10.3, $J_{6,7b}$=4.8 Hz, H-7b), 4.00 (d, 1 H, $J_{5,6}$=4.0 Hz, H-5), 4.22 (dt, 1 H, $J_{5,6}$=4.4, $J_{6,7b}$=4.5, $J_{6,7a}$=6.3 Hz, H-6), 4.92 (d, 1 H, $J_{3,4}$=4.8 Hz, H-4), 4.94 (m, 1 H, $J_{2a,3}$=2.2, $J_{2b,3}$=4.8, $J_{3,4}$=4.7 Hz, H-3). ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.0 (CH_3), 22.6, 25.7, 29.5, 31.6 (4 × CH_2 from side chain), 36.0 (C-2), 58.5 (OCH_3), 68.4 (C-7), 71.8 [$OCH_2(CH_2)_5CH_3$], 76.8 (C-3), 79.5 (C-6), 83.8 (C-5), 84.9 (C-4), 175.4 (C=O). HRMS-Heated ESI-Orbitrap: *m/z* 273.17019 (M^++H), calcd. for $C_{14}H_{25}O_5$: 273.1702; *m/z* 295.15213 (M^++Na), calcld. for $C_{14}H_{24}NaO_5$: 295.15214.

NMR SPECTRA OF FINAL PRODUCTS

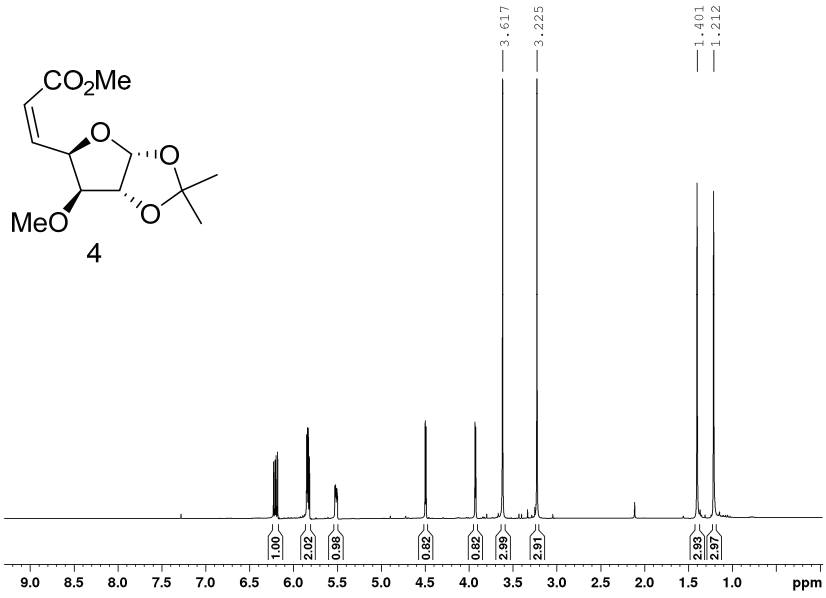

Fig. S-1. ^{1}H-NMR spectrum of **4** (400 MHz, $CDCl_3$).

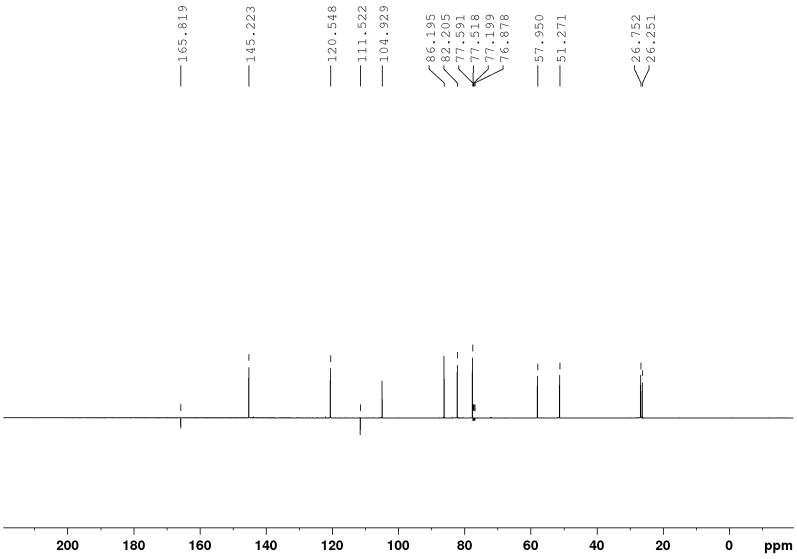

Fig. S-2. ^{13}C-NMR spectrum of **4** (100 MHz, $CDCl_3$).

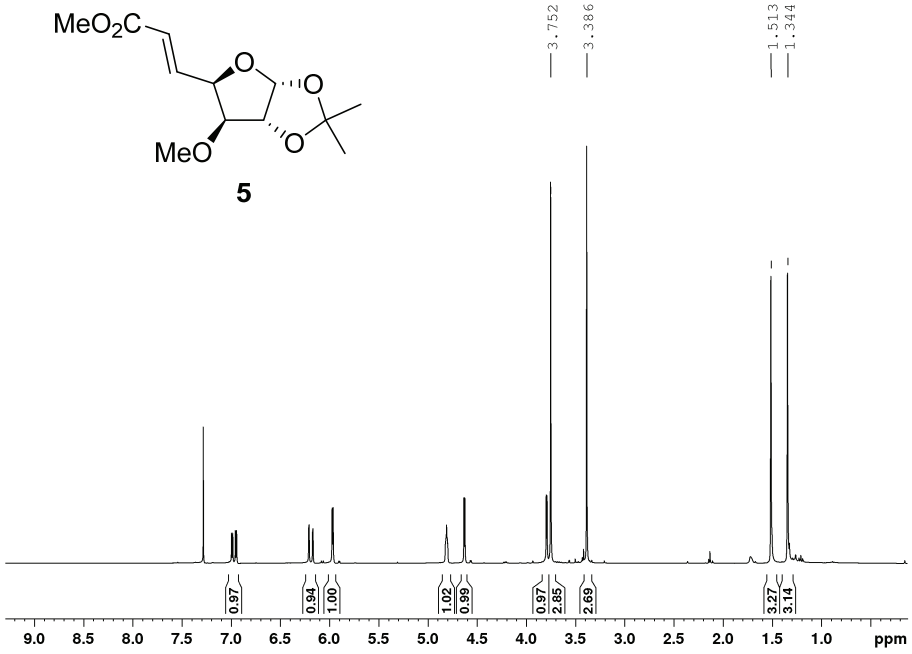

Fig. S-3. ^{1}H-NMR spectrum of **5** (400 MHz, $CDCl_3$).

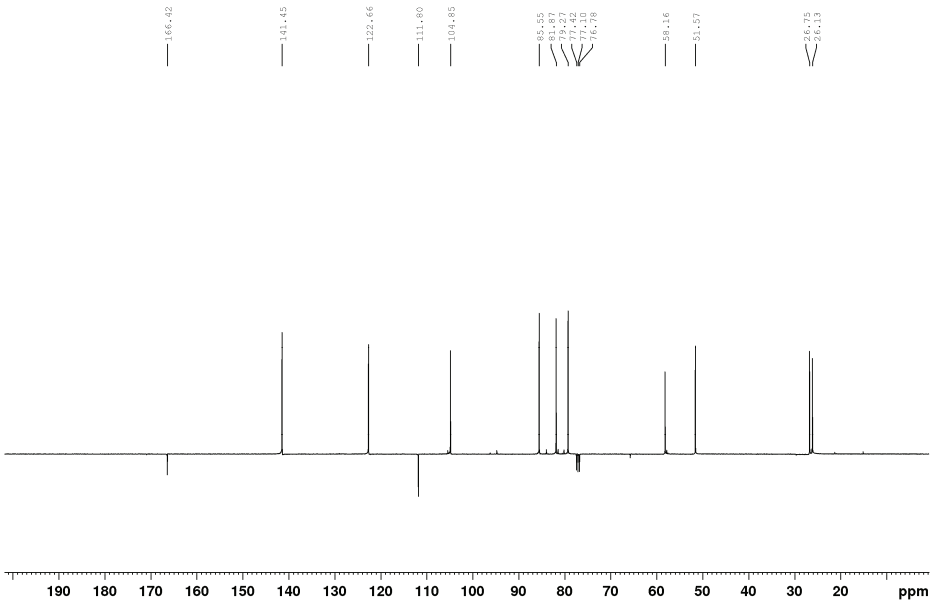

Fig. S-4. ^{13}C-NMR spectrum of **5** (100 MHz, $CDCl_3$).

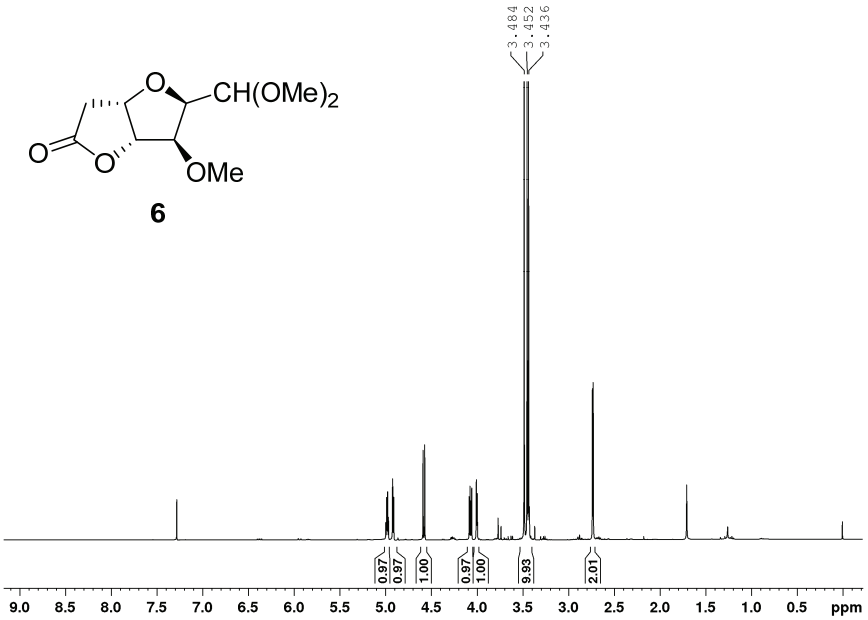

Fig. S-5. ^{1}H-NMR spectrum of **6** (400 MHz, $CDCl_3$).

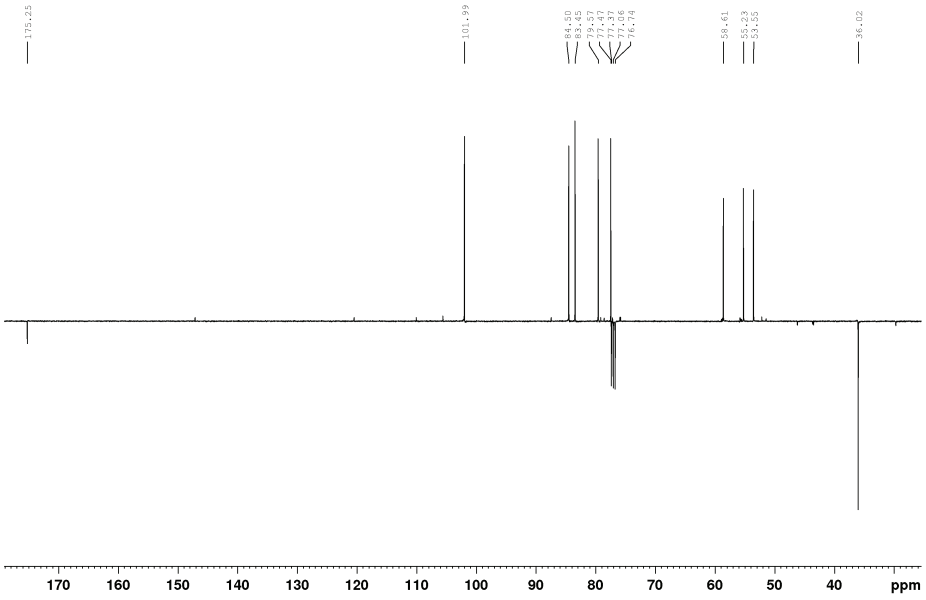

Fig. S-6. ^{13}C-NMR spectrum of **6** (100 MHz, $CDCl_3$).

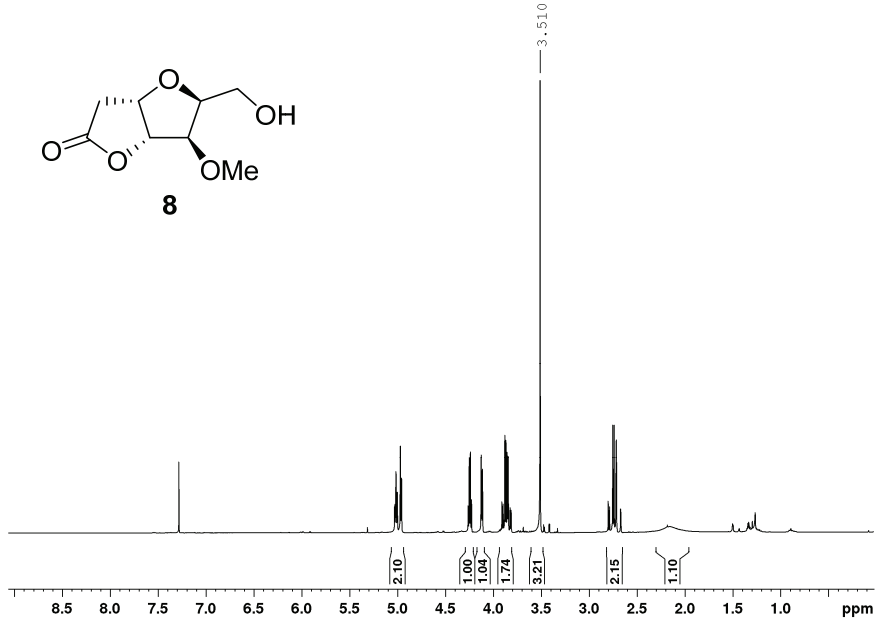

Fig. S-7. ^{1}H-NMR spectrum of **8** (400 MHz, $CDCl_3$).

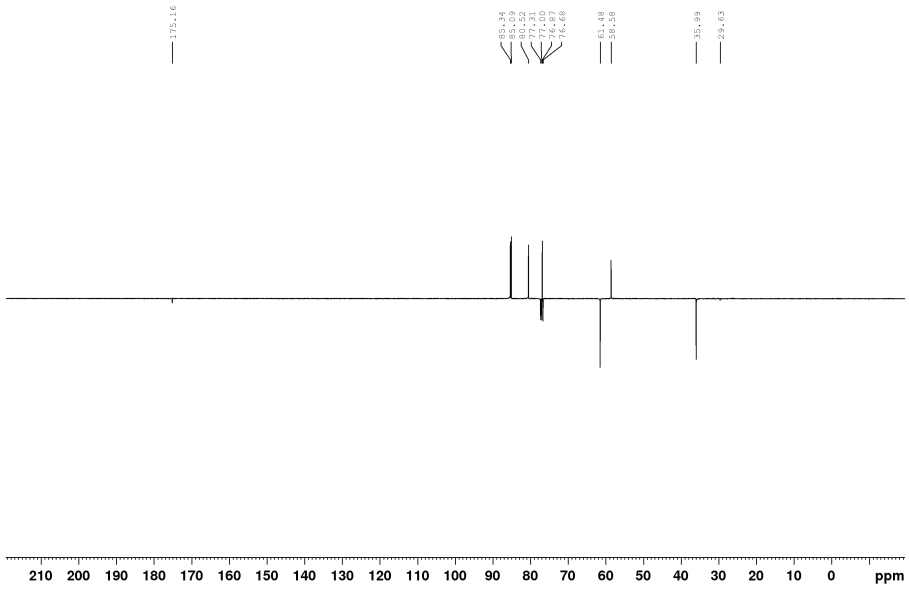

Fig. S-8. ^{13}C-NMR spectrum of **8** (100 MHz, $CDCl_3$).

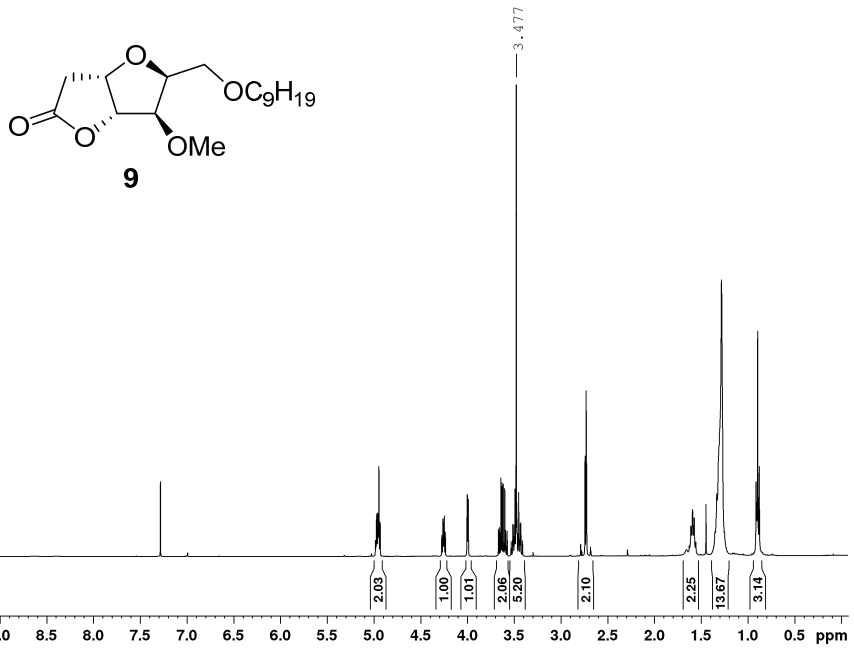

Fig. S-9. ^{1}H-NMR spectrum of **9** (400 MHz, $CDCl_3$).

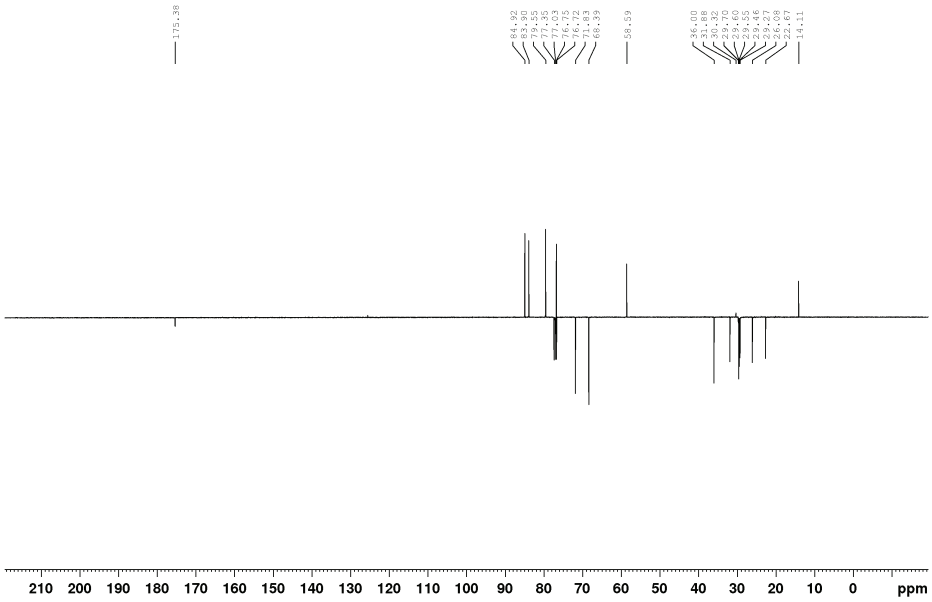

Fig. S-10. ^{13}C-NMR spectrum of **9** (100 MHz, $CDCl_3$).

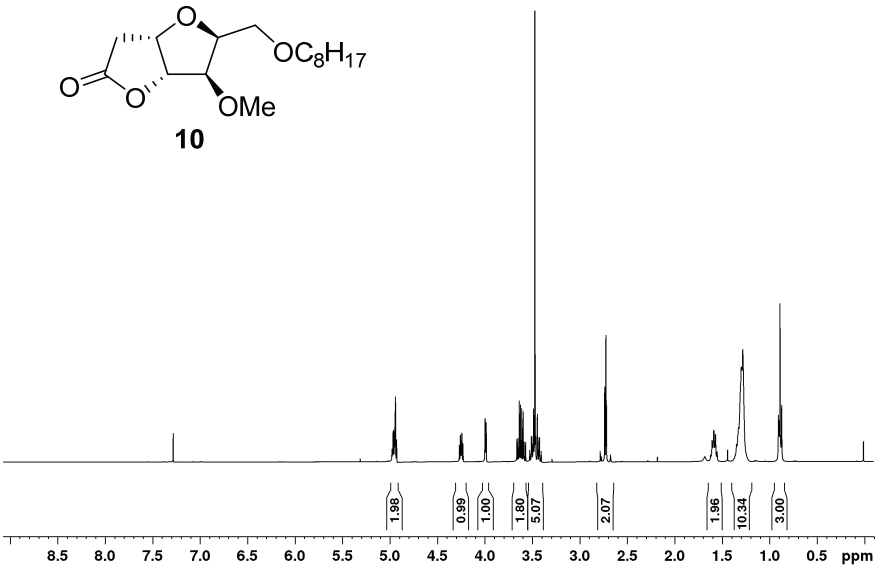

Fig. S-11. ^{1}H-NMR spectrum of **10** (400 MHz, $CDCl_3$).

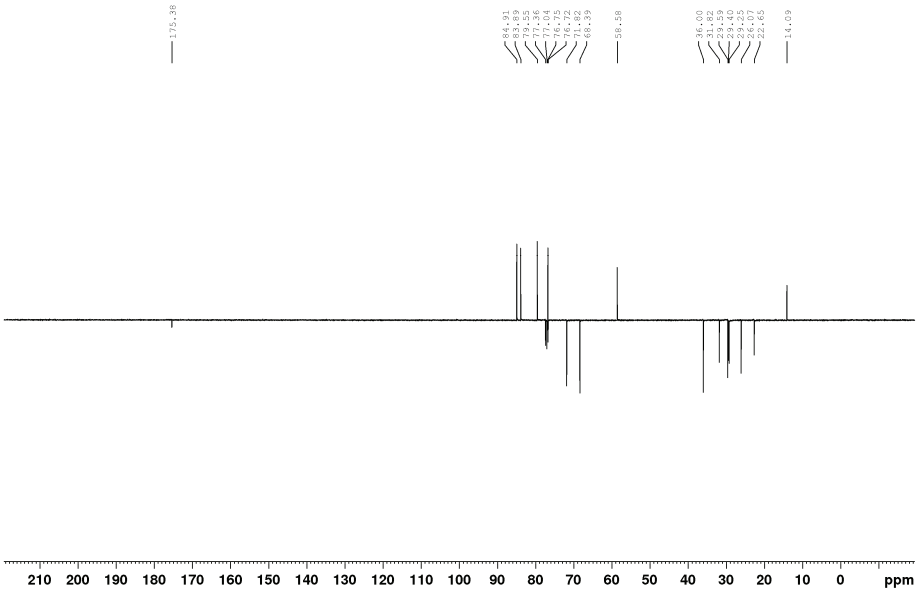

Fig. S-12. ^{13}C-NMR spectrum of **10** (100 MHz, $CDCl_3$).

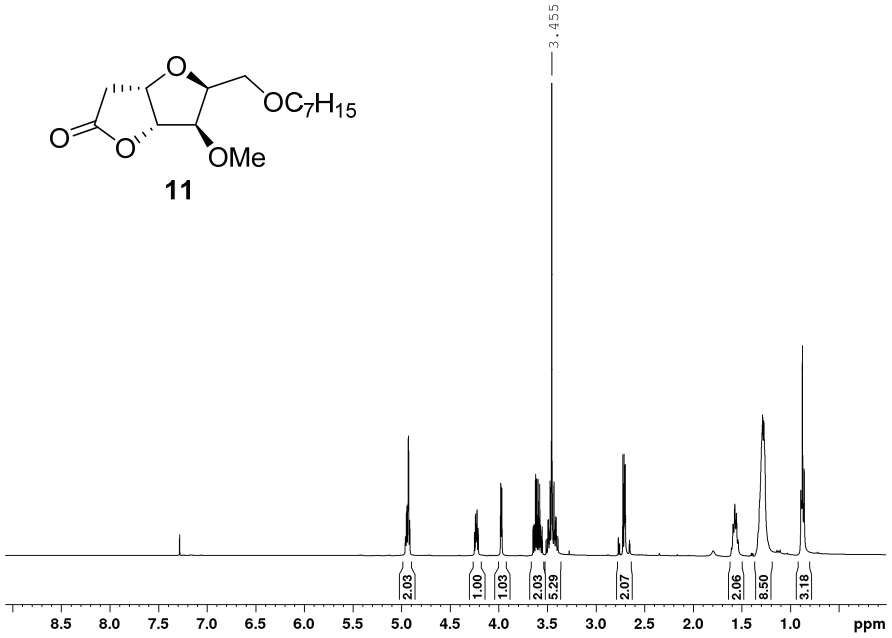

Fig. S-13. ^{1}H-NMR spectrum of **11** (400 MHz, $CDCl_3$).

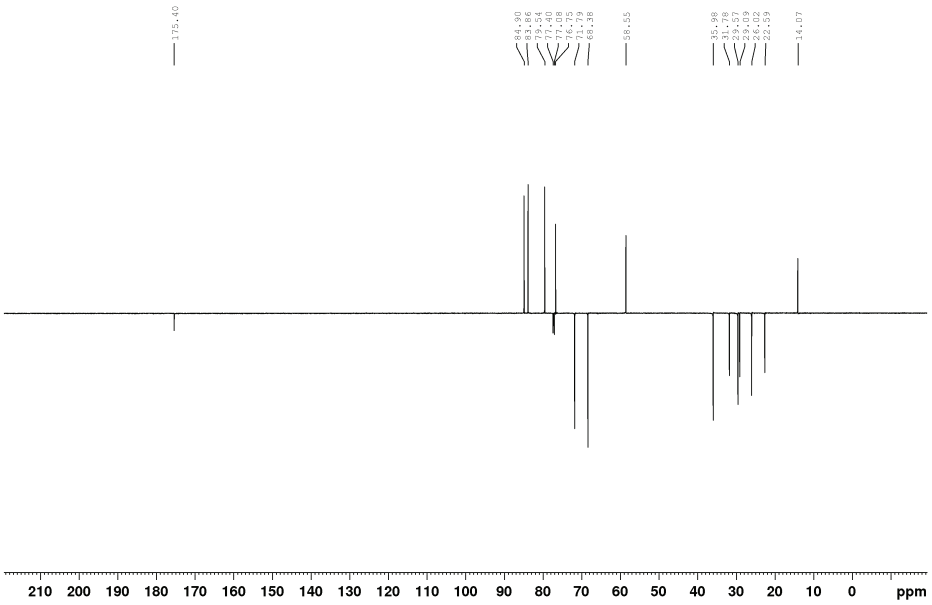

Fig. S-14. ^{13}C-NMR spectrum of **11** (100 MHz, $CDCl_3$).

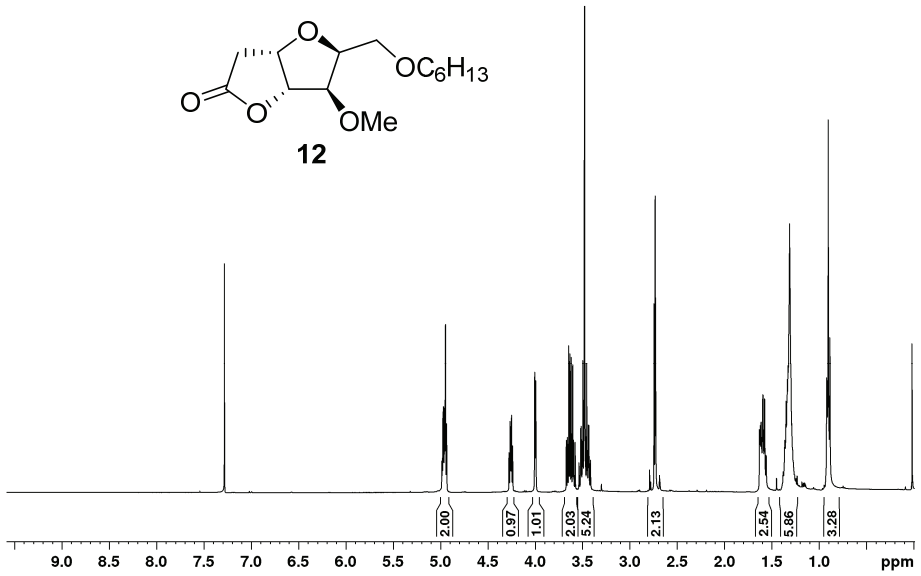

Fig. S-15. ^{1}H-NMR spectrum of **12** (400 MHz, $CDCl_3$).

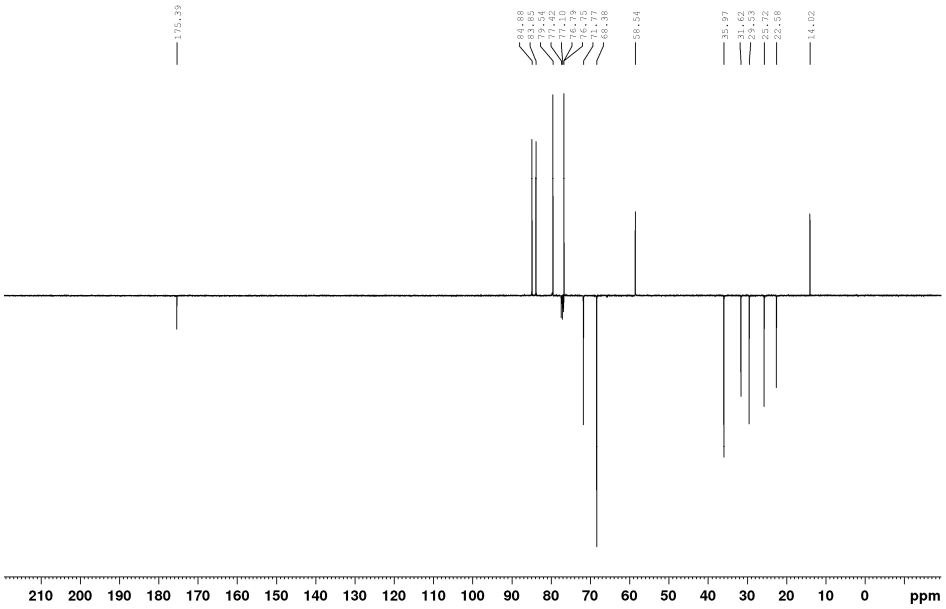

Fig. S-16. ^{13}C-NMR spectrum of **12** (100 MHz, $CDCl_3$).

SAR ANALYSIS

TABLE S-I. Cytotoxicity data for SAR analysis

Compound	K562	HL60	Jurkat	Raji	MCF-7	MDA-MB 231	HeLa
1	**0.04**	**25.85**	**100**	**0.1**	**21.35**	**100**	**0.17**
9	10.25	17.7	15.4	21.75	4.85	11.32	13.5
10	18.12	13.68	7.36	35.84	1.11	28.33	9.12
11	5.6	24.54	22.97	28.49	12.31	25.33	11.51
12	7.69	21.18	25.34	27.03	18.33	15.81	15.22
13	8.76	6.12	9.71	15.95	22.18	39.48	68.32
14	9.09	13.92	5.47	16.85	18.77	28.26	18.02
15	8.87	5.67	8.86	17.33	22.87	34.59	10.9
16	5.65	7.42	5.25	11.82	25.31	8.5	33.79
17[b]	5.66	4.75	6.97	7.25	102.36	296.78	6.39
18[b]	0.74	0.68	19.78	4.25	0.34	28.7	3.41
19[b]	1.02	1.1	11.53	5.98	2.38	9.76	0.56
20[b]	0.7	4.91	8.87	1.11	12.34	15.62	3.54

[a]IC_{50} is the concentration of compound required to inhibit the cell growth by 50% compared to an untreated control. Values are means of three independent experiments. Coefficients of variation were less than 10 %.
[b]Taken from reference[1]

The structure-activity relationships were accessed as follows: the IC_{50} values of two compounds were compared, and the Δ log IC_{50} was calculated (Δ log IC_{50} is a difference between the log IC_{50} values of an analogue and the corresponding control compound). Positive Δ log IC_{50} values show a decrease of antipro-

liferative activity, whereas negative values indicate an increase in the activity upon the structural modification being considered. The results are presented in Figure S-17.

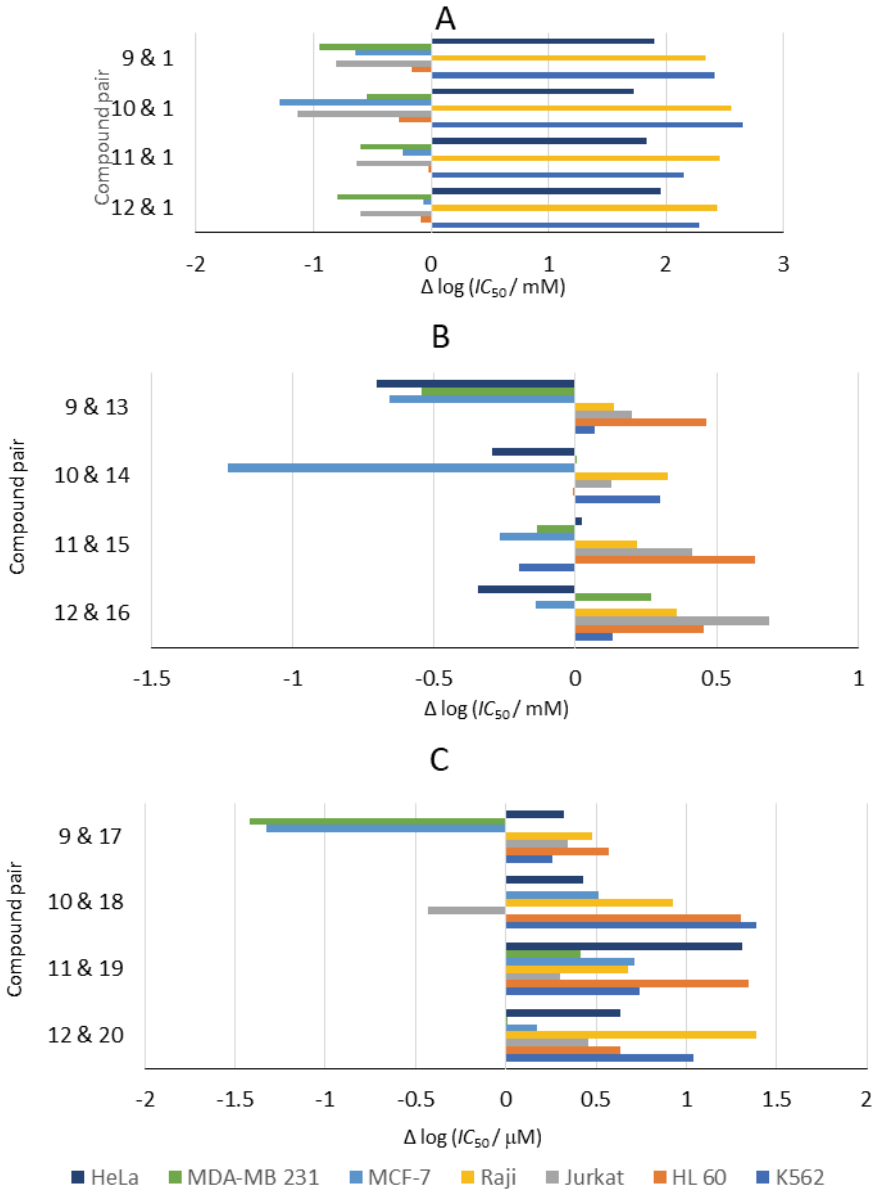

Fig. S-17. SAR Analysis. Influence of: (A) THF-ring closure, exchange of C_8 methylene group with O_8 ether function, 5-*O*-methylation; (B) substitution of methyl with benzyl group at C-5; (C) demethylation at C-5.

CRYSTAL STRUCTURE DETERMINATION

Diffraction experiments were performed on a Gemini S diffractometer. Data collection and reduction procedures were computed with CrysAlisPro.[2] Crystal structure was solved with SHEXLT,[3] and refined with SHEXL,[4] utilizing ShelXle[5] as a graphical user interface. The structure was validated internally by PLATON[6] and externally against Cambridge Structural Database[7] data by MOGUL[8] utility within Mercury CSD.[9] Crystallographic and refinement details are deposited in the Cambridge Crystallographic Data Centre under CCDC 2164235, obtainable free of charge from https://www.ccdc.cam.ac.uk/structures/. Selected crystallographic and refinement details are listed in Table S-II.

Table S-II. Pertinent crystallographic and refinement details of compound **8**

Crystal data	
Chemical formula	$C_8H_{12}O_5$
M_r	188.18
Crystal system	Orthorhombic
Space group	$P2_12_12_1$
Temperature, K	295
a / Å	7.4251 (2)
b / Å	7.7715 (2)
c / Å	16.2624 (5)
V / Å^3	938.41 (5)
Z	4
Radiation type	Mo $K\alpha$
μ / mm^{-1}	0.11
Crystal size, mm	0.58 × 0.36 × 0.28
Data collection	
Diffractometer	Gemini S (Oxford Diffraction)
Absorption correction	Multi-scan
T_{min}, T_{max}	0.918, 1.000
No. of measured	8304
No. of independent	2259
No. of observed [$I > 2\sigma(I)$] reflections	1989
R_{int}	0.021
$(\sin \theta / \lambda)_{max}$ / Å^{-1}	0.687
Refinement	
$R[F^2 > 2\sigma(F^2)]$	0.036
$wR(F^2)$	0.087
S	1.07
No. of reflections	2259
No. of parameters	123
H-atom treatment	Mixed
$\Delta\rho_{max}$, $\Delta\rho_{min}$ / e Å^{-3}	0.12, −0.18
Absolute structure parameter	Meaningless

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